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STUDY OF THE PHENOL RECOVERIES BY STEAM DISTILLATION

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Study of the Phenol Recoveries by Steam Distillation

MINISTRY OF THE ENVIRONMENT
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HAZARDOUS CONTAMINANTS OFFICE

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Ministry of the Environment

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STUDY OF THE PHENOL RECOVERIES BY STEAM DISTILLATION

INTRODUCTION

Phenols are hydroxy derivatives of aromatic hydrocarbons where the hydroxyl group is directly attached to the aromatic ring. Phenolic compounds are widely distributed in nature. The chemical industry produces millions of tons of various phenols as intermediates in the manufacture of a variety of products including dye, disinfectants, phenol-formaldehyde resins, epoxy resins, polycarbonates, pesticides, etc.

Of special environmental interest are the simple phenols containing one benzene ring. Owing to their relatively high water solubility simple phenols often enter surface and groundwater and can cause serious taste and odour problems.

While phenol as such can be detected in water by its odour or taste in concentrations of 0.01 - 0.1 mg/l, this effect is enhanced by 1-2 orders of magnitude, i.e. to 1 ug/L, upon chlorination for disinfection because of the formation of chlorophenols.

Gibbs and 4-aminoantipyrine are the two colourimetric methods used in our laboratory routinely to monitor phenols in water. To remove interferences, a distillation steps, which is outlined in the "Standard Methods" is used prior to both tests.

Recently, this laboratory has developed a sensitive direct aqueous injection (DAI) method for the determination of phenols using high performance liquid chromatography (HPLC) with a variable wavelength ultraviolet (UV) detector. This study was undertaken by using the DAI-HPLC method to determine recoveries of various phenols by the distillation technique.

PHENOL RECOVERY BY STEAM DISTILLATION

Ten phenols were evaluated for their ability to steam distill. Most of these compounds were listed in the United States Environmental Protection Agency's

"priority pollutants" list. They were phenol, o-chlorophenol, p-chlorophenol, p-cresol, m-chloro-p-cresol, 2,5-xylenol, 2,4-dichlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol and pentachlorophenol. Two distillation rates were used in this study. The effect of increased ionic strength in the water (salting-out) on recoveries was also investigated.

1. Phenol Solutions

Substrate	Solution 1	Solution 2
Phenol	300 ug/L	300 ug/L
p-cresol	317 ug/L	284 ug/L
o-chlorophenol	351 ug/L	-
p-chlorophenol	282 ug/L	338 ug/L
2,5-xylenol	281 ug/L	250 ug/L
m-chloro-p-cresol	336 ug/L	448 ug/L
2,4-dichlorophenol	272 ug/L	-
2,4,6-trichlorophenol	317 ug/L	-
2,4,5-trichlorophenol	294 ug/L	-
Pentachlorophenol	278 ug/L	-

2. Distillation

Two distillation rates were used, 0.2 mL/min and 0.5 mL/min.

A graduated Dean-Stark distilling receiver with stopcock was used to collect distillates for analysis.

3. HPLC

Analyses of samples were carried out by using a Varian 5000 liquid chromatograph equipped with a Varian MCH-10 reverse phase column. A Valco automatic loop injector using a 150 uL, or a 250 uL loop was used for injection. The eluent was a mixture of methanol and water. The water contained 0.5% (V/V) acetic acid. Solvent gradient was programmed from 50% to 80% methanol at 1%/min. The flow rate of the solvents was 1 mL/min. Individual phenols were detected and quantitated by using a Varian Varichrom variable wavelength UV detector. The wavelengths of the UV detector were set at 280 nm for phenol, o-chlorophenol, p-chlorophenol, p-cresol, 2,4-dichlorophenol, m-chloro-p-cresol, and 2,5-xylenol; 290 nm for 2,4,6-trichlorophenol and 2,4,5-trichlorophenol; and

300 nm for pentachlorophenol. Quantitation was achieved by comparison of chromatographic peaks with those of standards. The detection limit was 20 ug/L (250 uL injection), corresponding to 5 ng/injection.

4. Procedures

The phenol solution #1 or #2 (100 mL) was placed into a 250 mL round bottom flask. Phosphoric acid was added dropwise to adjust the acidity of the solution to approximately pH 3. Sodium chloride was added to solution #2 until the solution was saturated and few crystals of NaCl remained undissolved. A Dean-Stark distilling receiver was fitted between the flask and the condenser, and the distillation rate was set at 0.2 mL/min. The distillates were collected with the first three fractions containing approximately 1 mL each and the rest 5 mL each, producing a total of 13 fractions. The 13 distillation fractions and the residues were analyzed. The residues of solutions #2 were not analyzed because of their high salt content. The volumes of the distillates were measured according to the scale on the Dean-Stark distilling receiver, and were not corrected. The experiments were repeated with a distillation rate of 0.5 ml/min.

RESULTS AND DISCUSSION

The results of the recoveries of phenols by steam distillation were tabulated and diagrams were prepared for illustration.

From the data collected, it was observed that each individual phenol had its own characteristic steam distillation pattern. The pattern was dependent on the number, position, and type of functional groups that make up the individual phenol. Distillation rates had some influence on the yield of recoveries, especially at the beginning of the distillation. However, they showed the same general trend in distillation pattern. Salt played a very significant role, it produced faster and higher recoveries.

Phenol

Phenol was steam distilled over at an even, moderate rate. After 55 mL was distilled over from the 100 mL solution, phenol was only partially recovered in the distillate.

At 0.5 mL/min distillation rate, phenol concentrations in the early fractions were slightly higher than those in the later fractions. The concentrations were 600 ug/L at the beginning and 200 ug/L in the last 5 mL fraction collected. The phenol concentration in the remaining solution (residue) was 93 ug/L. The recovery of phenol in the first 30 mL distillate was 55.2% and in the total distillate was 84.4%.

At 0.2 mL/min distillation rate, phenol concentrations in the early fractions were much higher than those in the later fractions. The phenol concentrations in the first 15 mL was in the 600 to 1100 ug/L range. The concentrations in the later fractions were very similar to those in the 0.5 mL/min distillation. The phenol concentration in the residue was 62 ug/L. The phenol recovery in the first 30 mL distillate was 65.1% and in the total distillate was 88.5%.

When the phenol solution was saturated with NaCl, phenol distilled over at a faster rate. After 15 mL distilled over, the phenol concentration in the subsequent distillates was beyond detection limit. The recovery was slightly higher when distillation rate was slower. The total recovery was 72.4% at 0.2 mL/min rate and 66.7% at 0.5 mL/min. See Figure 1.

p-Cresol

p-Cresol steam distilled slightly faster than phenol. After 55 mL of distillate was collected, p-cresol was not detected in the residue. The distillation patterns of p-cresol were similar to those of phenol, except that p-cresol had higher concentrations in the distilled fractions and recoveries were higher.

At 0.5 mL/min distillation rate, the p-cresol concentration was maintained at approximately 900 ug/L at the beginning of the distillation then it diminished slowly. The recovery in the first 30 mL of distillate collected was 72.9%. The total recovery was 97.2%.

At 0.2 ml/min distillation rate, the p-cresol concentration was higher at the beginning ranged from 1000 to 2000 ug/L, then it fell to the same level as that observed at 0.5 mL/min distillation. The recovery in the first 30 mL distillate was 86.5% and the total recovery was 103.7%.

When the solution was saturated with NaCl, p-cresol distilled over at a much faster rate. p-cresol was only detected in the first 15 mL distillate. The total recovery was 70% for both distillation rates. See Figure 2.

o-Chlorophenol

o-Chlorophenol steam distilled at very fast rate. After 55 ml of water distilled over from a 100 ml solution, o-chlorophenol was not detected in the residue.

At 0.5 ml/min distillation rate, o-chlorophenol was detected in the first 30 mL of distillates. The concentration was more than 6000 ug/L at the beginning, then diminished rapidly. The total recovery for 30 mL distillate was 105.1%.

At 0.2 mL/min distillation rate, o-chlorophenol steam distilled faster. It was not detected after the first 10 mL of distillate was collected. The concentration of the first fraction collected was over 7000 ug/L. The total recovery from the 10 mL distillate collected was 90.3%. See Figure 3.

p-Chlorophenol

p-Chlorophenol steam distilled slower than phenol. After 55 mL was distilled over from a 100 mL solution, p-chlorophenol was still detected in the residue.

At 0.5 mL/min distillation rate, the concentration of p-chlorophenol in each distillation fraction remained in the 300-400 ug/L range. The recovery of p-chlorophenol in the first 30 mL distillate was 38.7%. The recovery for the entire 55 mL distillate collected was 67.8%. The concentration of p-chlorophenol in the residue was 219 ug/L.

At 0.2 mL/min distillation rate, the concentrations of p-chlorophenol were slightly higher in the first 15 mL of distillates. However, the concentrations of most fractions remained in the 300-400 ug/L range. The recovery in the first 30 mL distillate was 41.5%. The recovery in the entire distillate was 70.5%. The concentration in the residue was 164 ug/l. Similar distillation patterns were observed at both distillation rates.

When the solution was saturated with NaCl, p-chlorophenol steam distilled at a faster rate.

The concentration of p-chlorophenol in the distillation fractions were high at the beginning and then tapered off rapidly. This was different from distillation pattern observed without saturating the solution with NaCl.

At 0.5 mL/min distillation rate, p-chlorophenol was detected in the first 30 mL distillates collected. The recovery was 64.3%. At 0.2 mL/min distillation rate, it was detectable in the first 20 mL distillate. The recovery was 76.6%. The distillation patterns with these two distillation rates were similar. See Figure 4.

2,5-Xylenol

2,5-Xylenol steam distilled faster than p-cresol but slower than o-chlorophenol. After 55 ml was distilled over from a 100 mL solution, no 2,5-xylenol was detected in the residue when the distillation rate was at 0.2 mL/min, and 27 ug/L was detected at the rate of 0.5 mL/min.

At 0.5 ml/min distillation rate, 2,5-xylenol distilled over at concentration ranges from 800 to 1900 ug/L in the first 10 mL. The last 5 mL of the 55 mL collected contained 27 ug/L. Ther recovery in the first 30 mL was 93.5%. The recovery in the total distillate was 98.8%.

At 0.2 mL/min distillation rate, 2,5-xylenol distilled over very fast at the beginning. The concentrations of the fractions collected in the first 10 mL ranged between 1200 ug/L and 3400 ug/L. After 35 mL was distilled over, no more 2,5-xylenol was detected in the subsequent fractions.

When the solution was saturated with NaCl, 2,5-xylenol steam distilled over at a faster rate. The first distillation fraction (1.0 mL) collected had the highest concentration, 12,000 ug/L, then the concentrations in the subsequent fractions tapered off rapidly. After 15 mL of distillate was collected, no 2,5-xylenol was detected in the later fractions. The distillation patterns were similar when distilled at 0.2 mL/min and 0.5 mL/min rates. The recoveries were 76.8% and 73.9% respectively. See Figure 5.

m-Chloro-p-cresol

The distillation patterns and recoveries for m-chloro-p-cresol were similar to those of phenol.

At 0.5 mL/min distillation rate, the concentrations of m-chloro-p-cresol in most of the distillation fractions remained between 500 ug/L and 700 ug/L. Only in the last 4 fractions were the concentrations below 500 ug/L. The concentration in the residue was 98 ug/L. The recovery of m-chloro-p-cresol in the first 30 mL was 58.4% and the total recovery was 88.8%.

At 0.2 mL/min distillation rate, the concentration in the fractions collected at the beginning of the distillation was over 900 ug/L and the rest were similar to those distilled at 0.5 mL/min rate. The concentration in the residue was 54 ug/L. The recovery of m-chloro-p-cresol in the first 30 mL was 71.7% and the total recovery was 97%.

When the solution was saturated with NaCl, m-chloro-p-cresol steam distilled over at a faster rate. After 15 mL of distillate was collected, no more 2,5-xylenol was detected in the later fractions. The recoveries were 71.3% at 0.5 ml/min distillation rate and 72.8% at 0.2 mL/min rate. See Figure 6.

2,4-Dichlorophenol

2,4-Dichlorophenol steam distilled faster than 2,5-xylenol but slower than o-chlorophenol. The distillation patterns were very similar to those for o-chlorophenol. After 55 mL was distilled over from a 100 mL, 272 ug/L solution, no 2,4-dichlorophenol was detected in the residue. At 0.5 mL/min distillation rate, the concentrations in the fractions collected up to 5 mL were between 2000 ug/L and 4000 ug/L. After 30 mL of distillate was collected, no 2,4-dichlorophenol was detected in the subsequent fractions. At 0.2 mL/min distillation rate, the recovery rate was much faster, the concentrations of the fractions collected up to 5 mL were between 2000 ug/L and 6200 ug/L. All 2,4-dichlorophenol was recovered in the first 15 mL distillate. See Figure 7.

2,4,6-Trichlorophenol

2,4,6-Trichlorophenol steam distilled faster than 2,5-xylenol but slower than 2,4-dichlorophenol. After 55 mL was distilled over from a 100 mL, 317 ug/L

solution, no 2,4,6-trichlorophenol was detected in the residue. At 0.5 mL/min distillation rate, the concentrations of the fractions up to the first 5 mL were between 2500 ug/L and 4500 ug/L. After 30 mL of distillate was collected, no 2,4,6-trichlorophenol was detected in the subsequent fractions. At 0.2 mL/min distillation rate, the concentrations of the first three fractions were very high; they ranged from 2500 ug/L to 8500 ug/L and 2,4,6-trichlorophenol distilled over in the first 20 mL of distillate. See Figure 8.

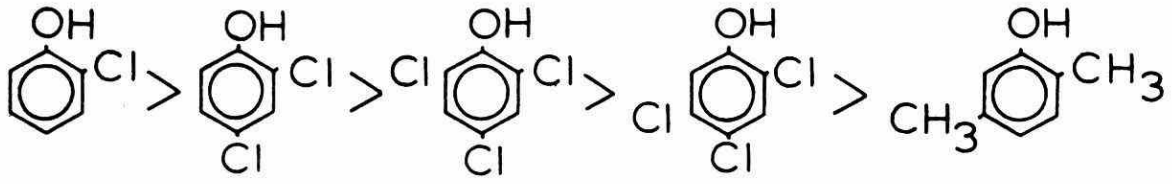
2,4,5-Trichlorophenol

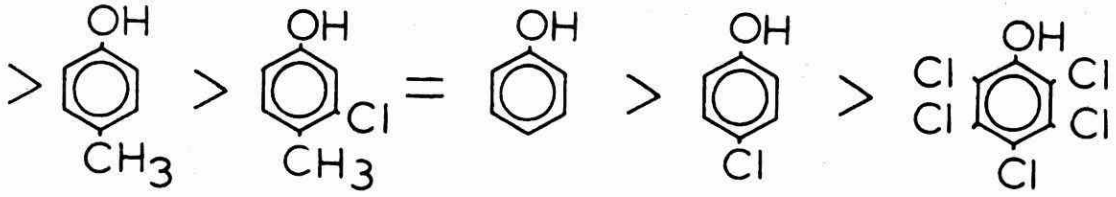
2,4,5-trichlorophenol steam distilled faster than 2,4-xylenol and slower than 2,4,6-trichlorophenol. The distillation patterns were very similar to those of 2,4,6-trichlorophenol. At 0.5 mL/min distillation rate, the concentration of 2,4,5-trichlorophenol in the fractions collected up to the first 10 mL were between 1000 ug/L and 2500 ug/L. It was recovered within the first 40 mL of distillate. At 0.2 mL/min, the concentrations were between 1000 ug/L to 5000 ug/L and the substrate was recovered within the first 20 mL. See Figure 9.

Pentachlorophenol

Pentachlorophenol steam distilled slower than p-chlorophenol and it was the slowest one among the ten phenols tested. After 55 mL distilled over from a 100 mL 278 ug/L solution, most of the Pentachlorophenol remained in the residue. The concentrations of Pentachlorophenol in the distillation fractions were lower than in the original solution. It was also interesting to notice that the concentrations in the distillation fractions remained constant, and faster distillation gave slightly higher yield. See Figure 10.

From the results obtained, the relative recoveries of the ten phenols by steam distillation can be summarized as follows:





It was also observed that in most cases, slower distillation rates gave higher recoveries. There were exceptions: p-chlorophenol showed little differences and pentachlorophenol showed a reverse order.

By saturating the solution with NaCl before distillation, the concentrations of phenols in the distillation fractions collected at the beginning were significantly higher compared to those without NaCl. In most cases, phenols were detected in the first 15 mL distillates. Because of high salt content in the residues, their phenols concentrations were not analyzed.* There were no significant differences observed in the total recoveries and distillation patterns when distilled at different distillation rates. The recoveries for the phenols studied were in the 70%-80% range. Large quantities of salt crystals formed during distillation; it was followed with severe bumping, especially when distilled at higher temperature. This may explain the low recoveries observed in this experiment. By using lower concentrations of NaCl, bumping may be avoided during distillation, thus the salting-out should be optimised.

CONCLUSION

The recoveries of ten phenols were studied by steam distillation. The recoveries were dependent on the number, position and type of substitutions on the phenol, and to a lesser extent, on the distillation rate. By adding NaCl to the solution before distillation, the recoveries improved and the role played by the substitution groups on phenol diminished. Steam distillation, especially with added NaCl, is a useful technique for concentrating and cleaning up phenol samples, but recoveries do vary with individual phenols and these should be taken into account when evaluating analytical results.

* NB: The "salt cakes" should have been extracted, evaporated, etc. and checked for trapping of phenols.

Appendices

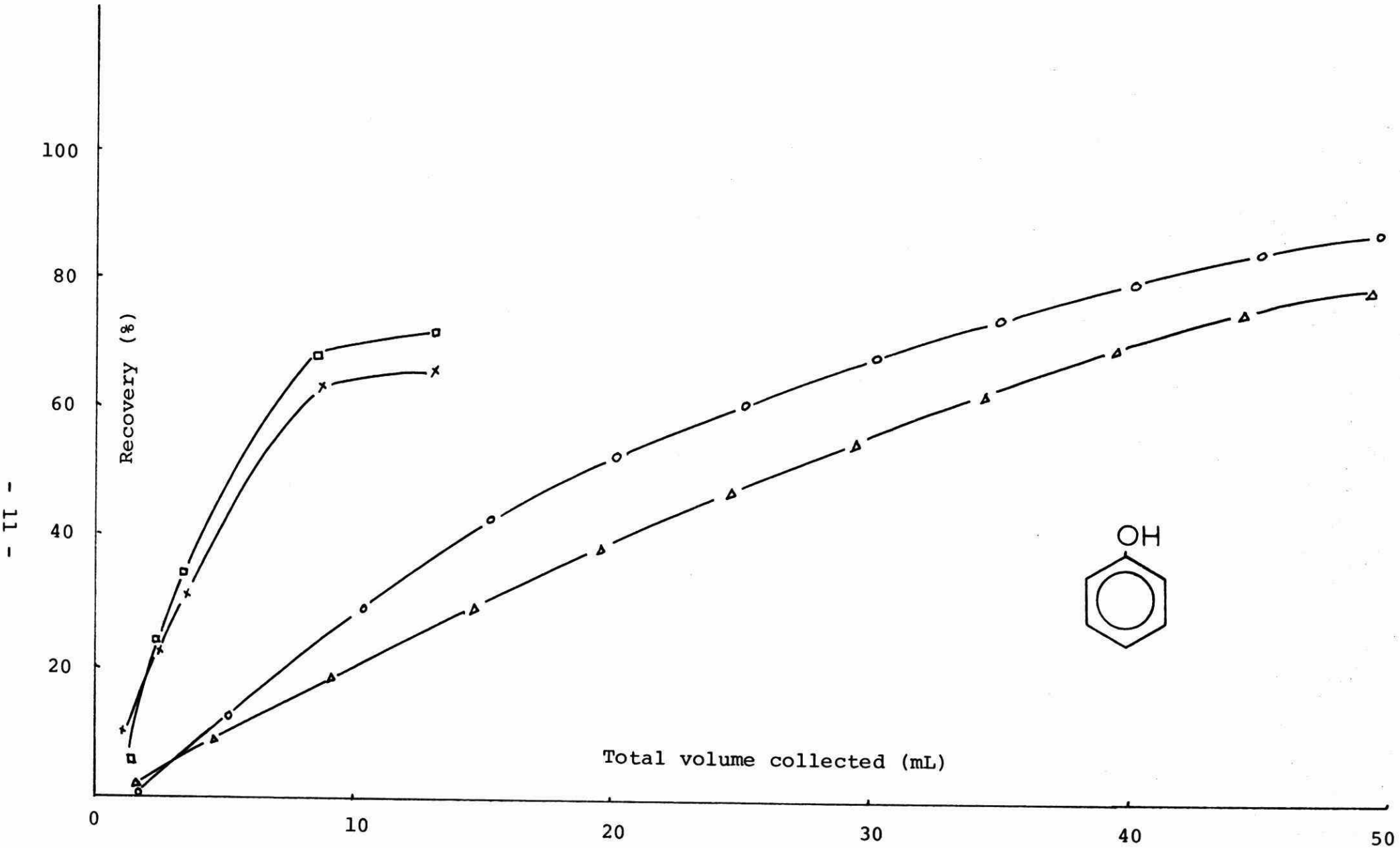


Figure 1. The recovery of phenol by steam distillation of a 100 mL solution
- ▫ Distilled at 0.2 mL/min rate, saturated with salt
- × Distilled at 0.5 mL/min rate, saturated with salt
- ○ Distilled at 0.2 mL/min rate, no salt
- △ Distilled at 0.5 mL/min rate, no salt

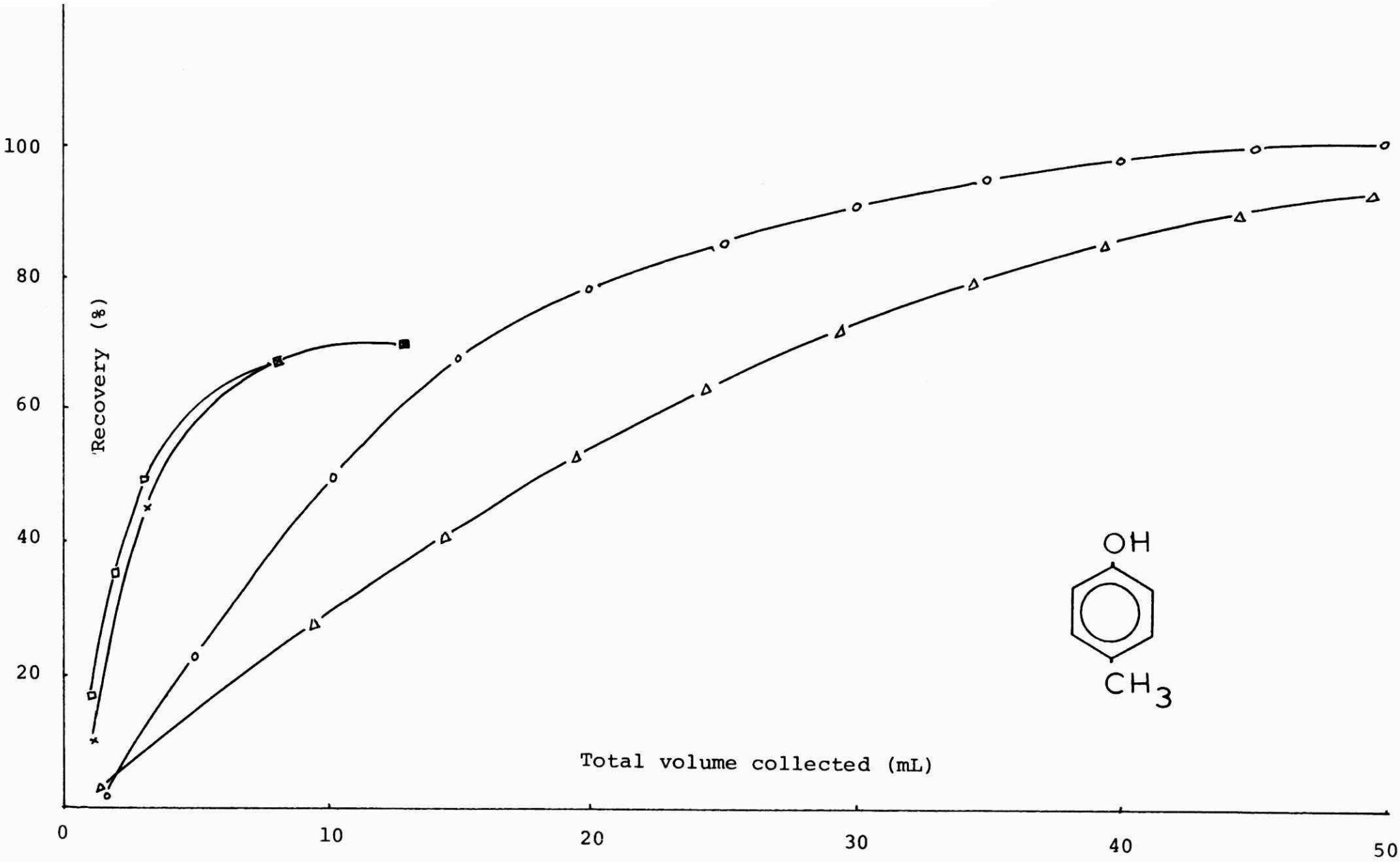


Figure 2. The recovery of p-cresol by steam distillation of a 100 mL solution

- ▫ Distilled at 0.2 mL/min rate, saturated with salt
- × Distilled at 0.5 mL/min rate, saturated with salt
- ○ Distilled at 0.2 mL/min rate, no salt
- △ Distilled at 0.5 mL/min rate, no salt

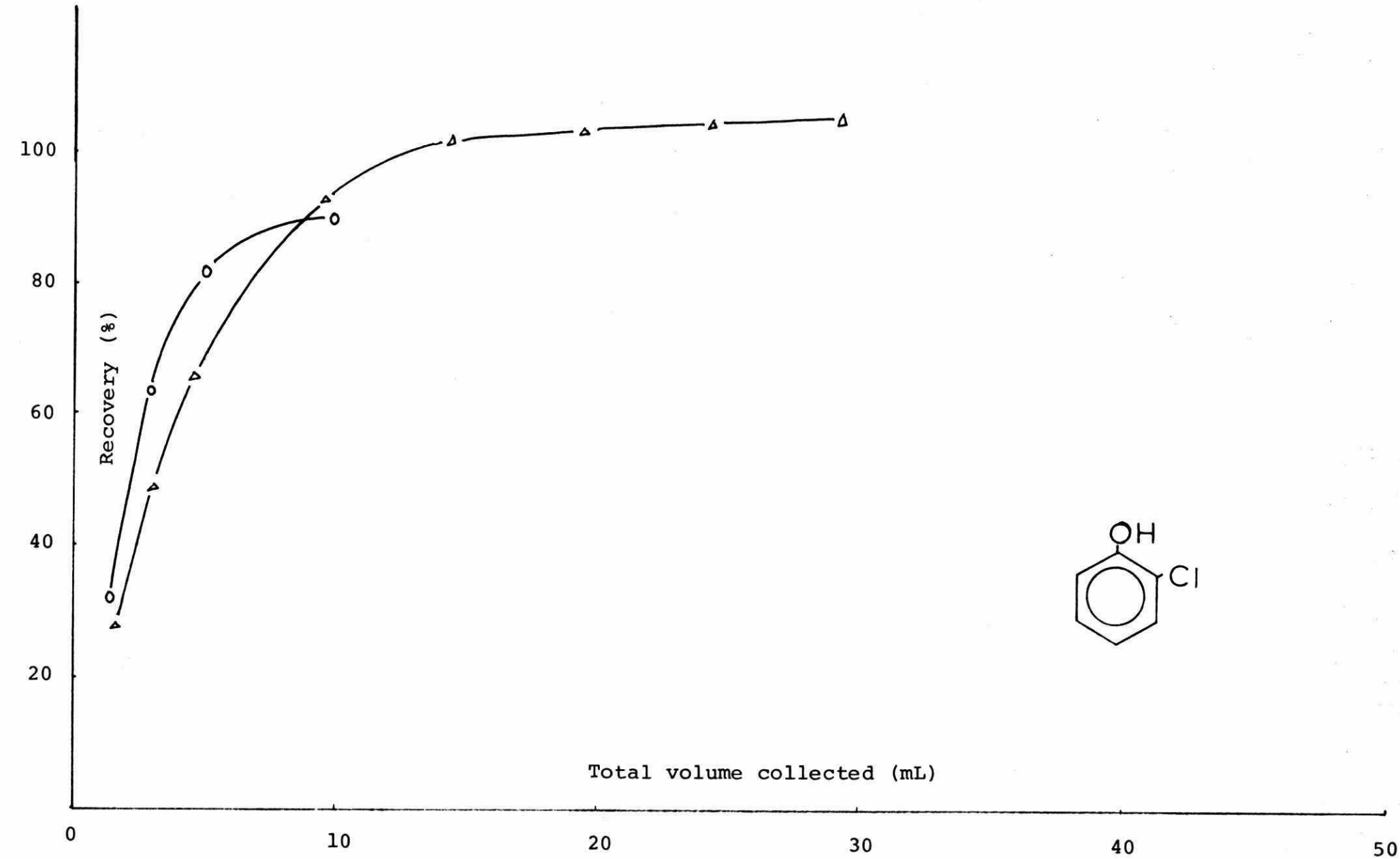


Figure 3. The recovery of o-chlorophenol by steam distillation of a 100 mL solution
o Distilled at 0.2 mL/min rate, no salt
Δ Distilled at 0.5 mL/min rate, no salt

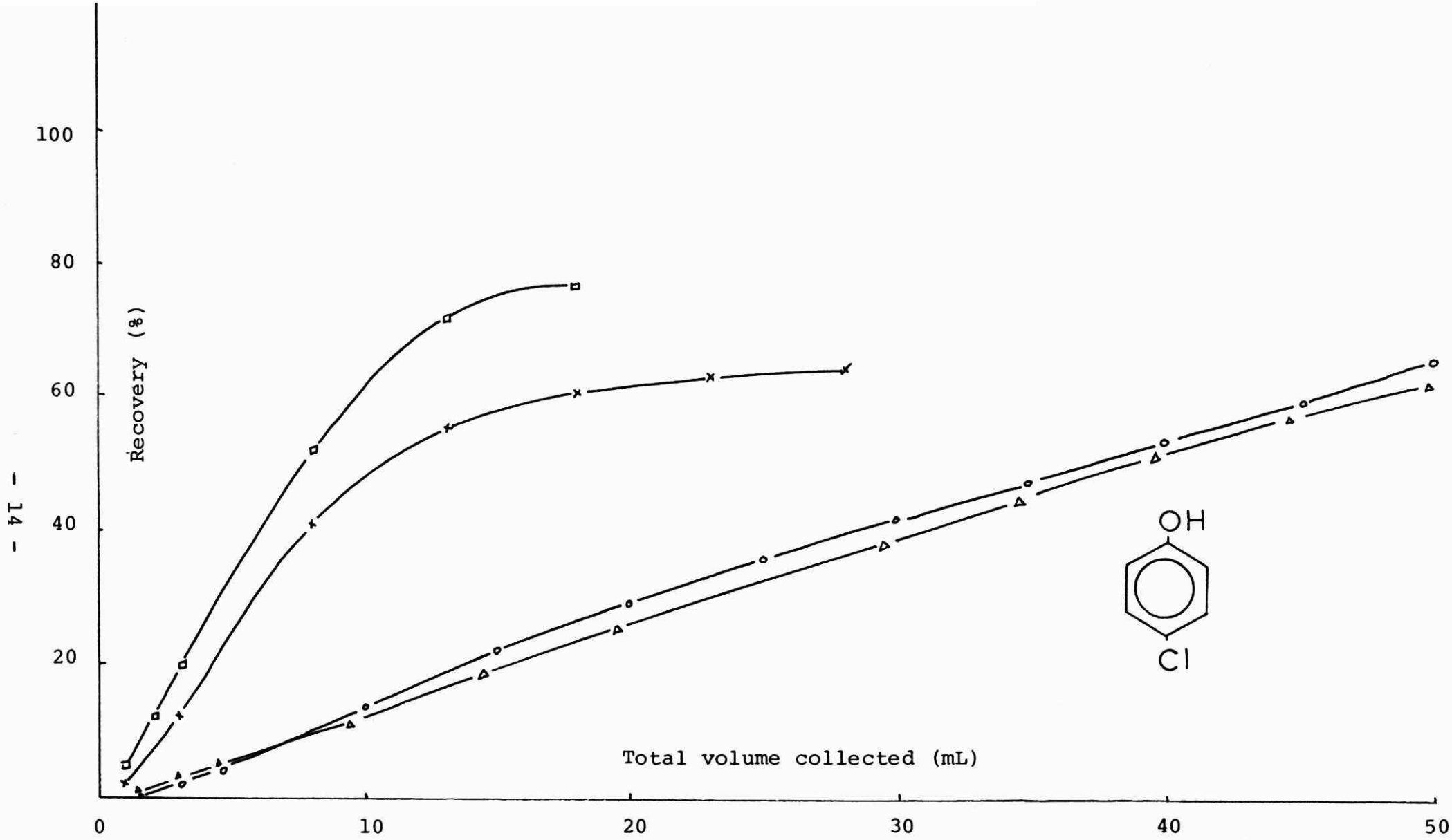


Figure 4. The recovery of p-chlorophenol by steam distillation of a 100 mL solution
- ▫ Distilled at 0.2 mL/min rate, saturated with salt
- × Distilled at 0.5 mL/min rate, saturated with salt
- ○ Distilled at 0.2 mL/min rate, no salt
- △ Distilled at 0.5 mL/min rate, no salt

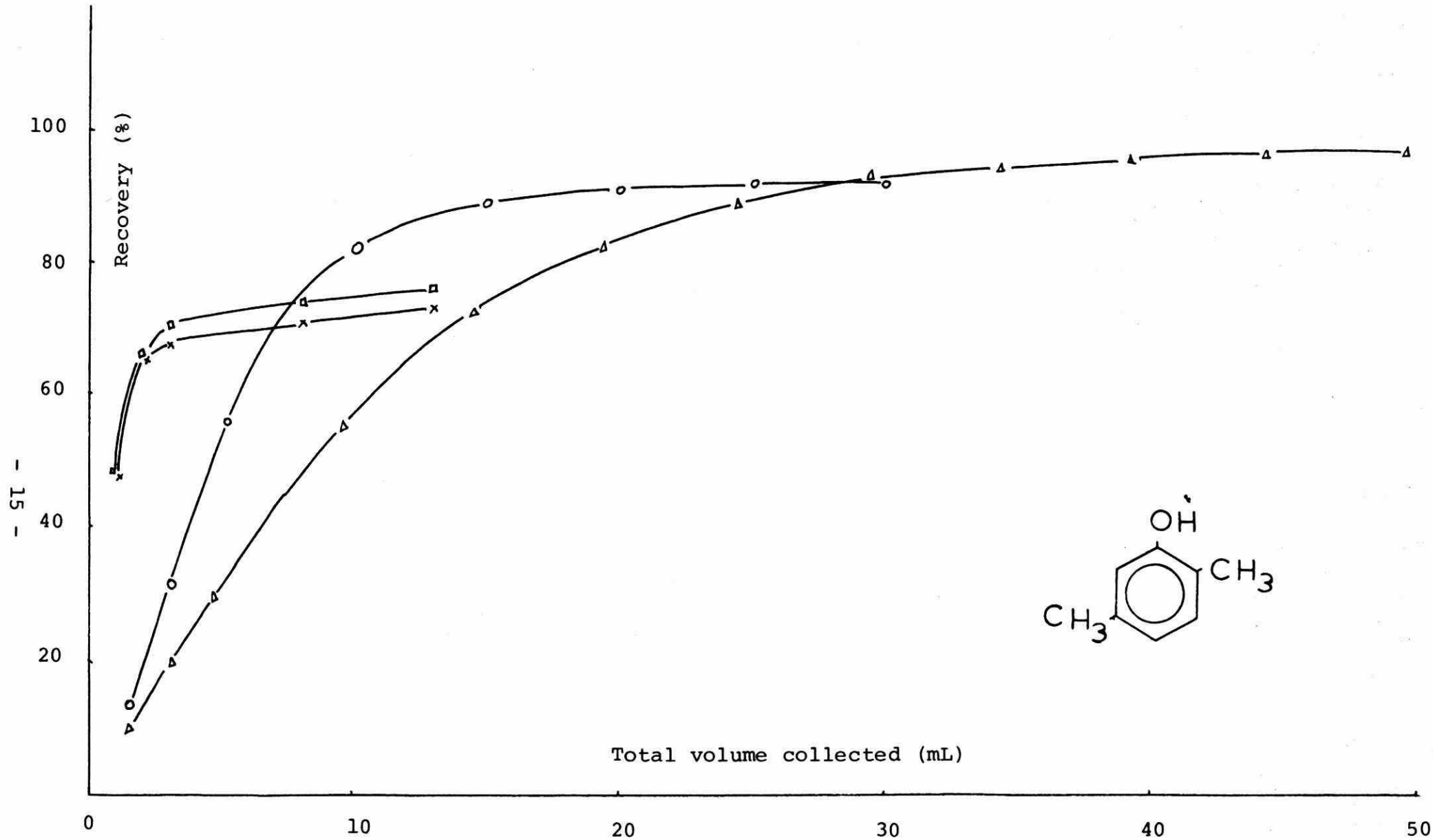


Figure 5. The recovery of 2,5-xylenol by steam distillation of a 100 mL solution
- ▫ Distilled at 0.2 mL/min rate, saturated with salt
- x Distilled at 0.5 mL/min rate, saturated with salt
- o Distilled at 0.2 mL/min rate, no salt
- △ Distilled at 0.5 mL/min rate, no salt

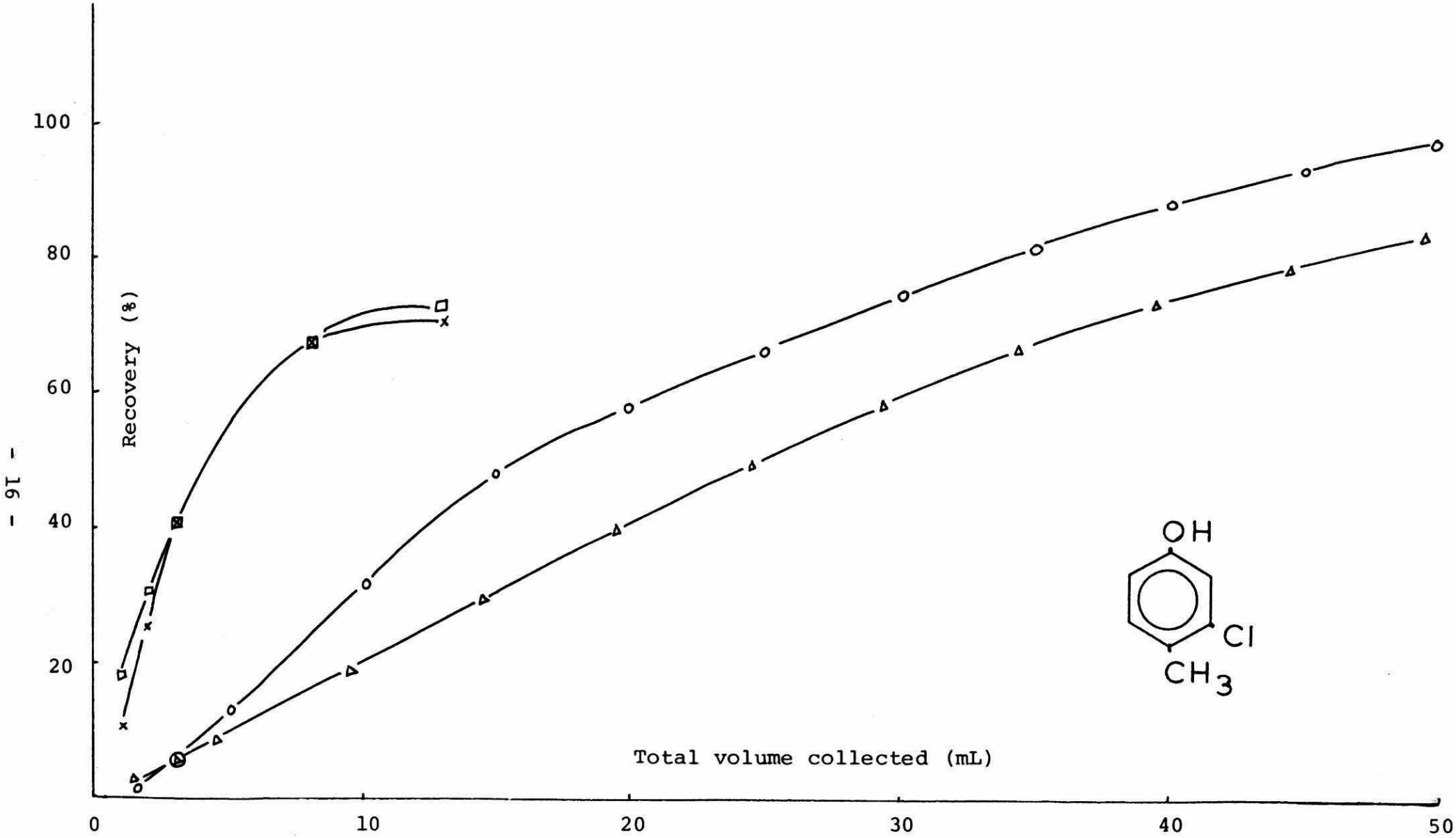


Figure 6. The recovery of m-chloro-p-cresol by steam distillation of a 100 mL solution

- ▫ Distilled at 0.2 mL/min rate, saturated with salt
- x Distilled at 0.5 mL/min rate, saturated with salt
- ○ Distilled at 0.2 mL/min rate, no salt
- △ Distilled at 0.5 mL/min rate, no salt

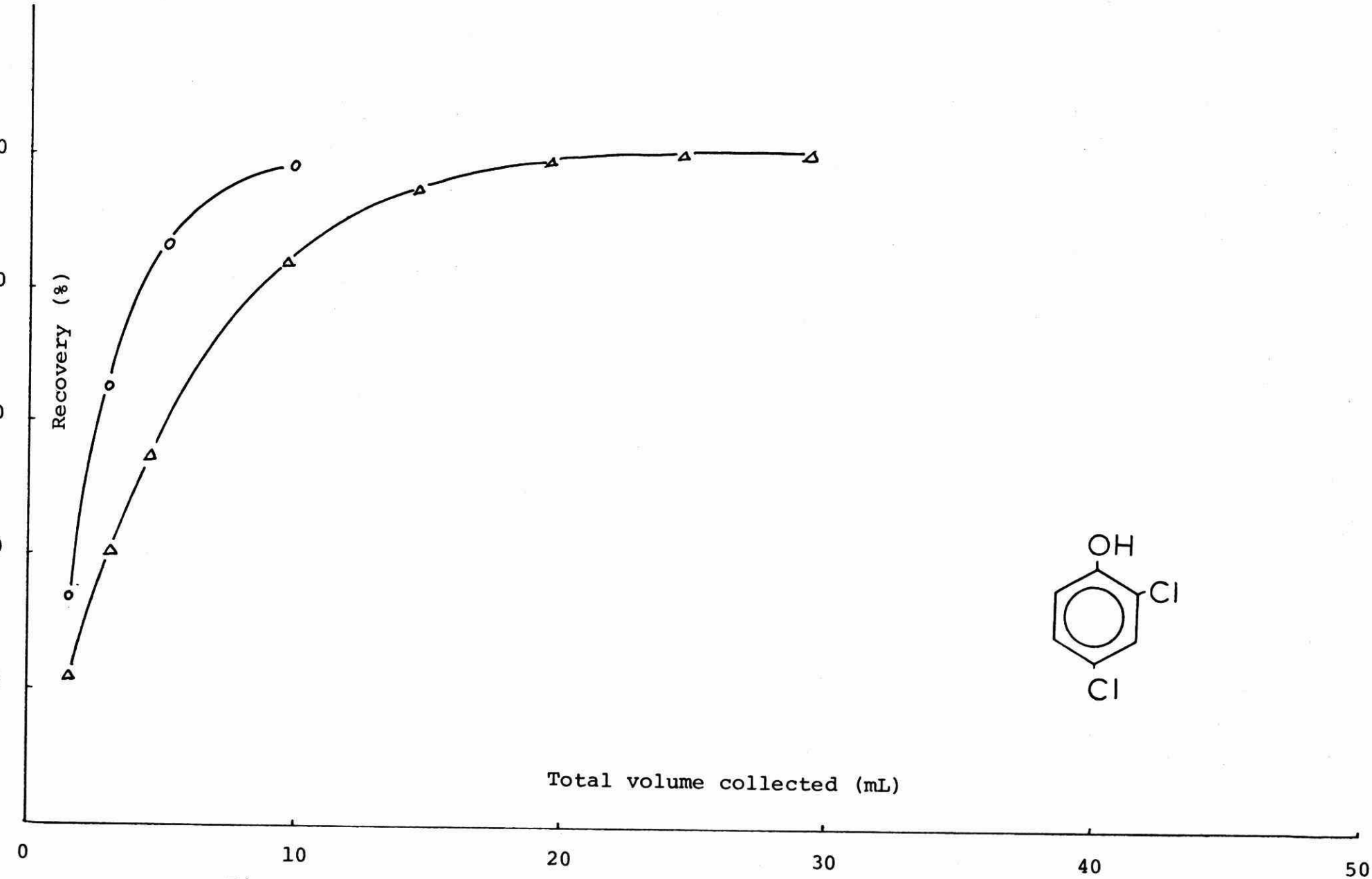


Figure 7. The recovery of 2,4-dichlorophenol by steam distillation of a 100 mL solution
o Distilled at 0.2 mL/min rate, no salt
▲ Distilled at 0.5 mL/min rate, no salt

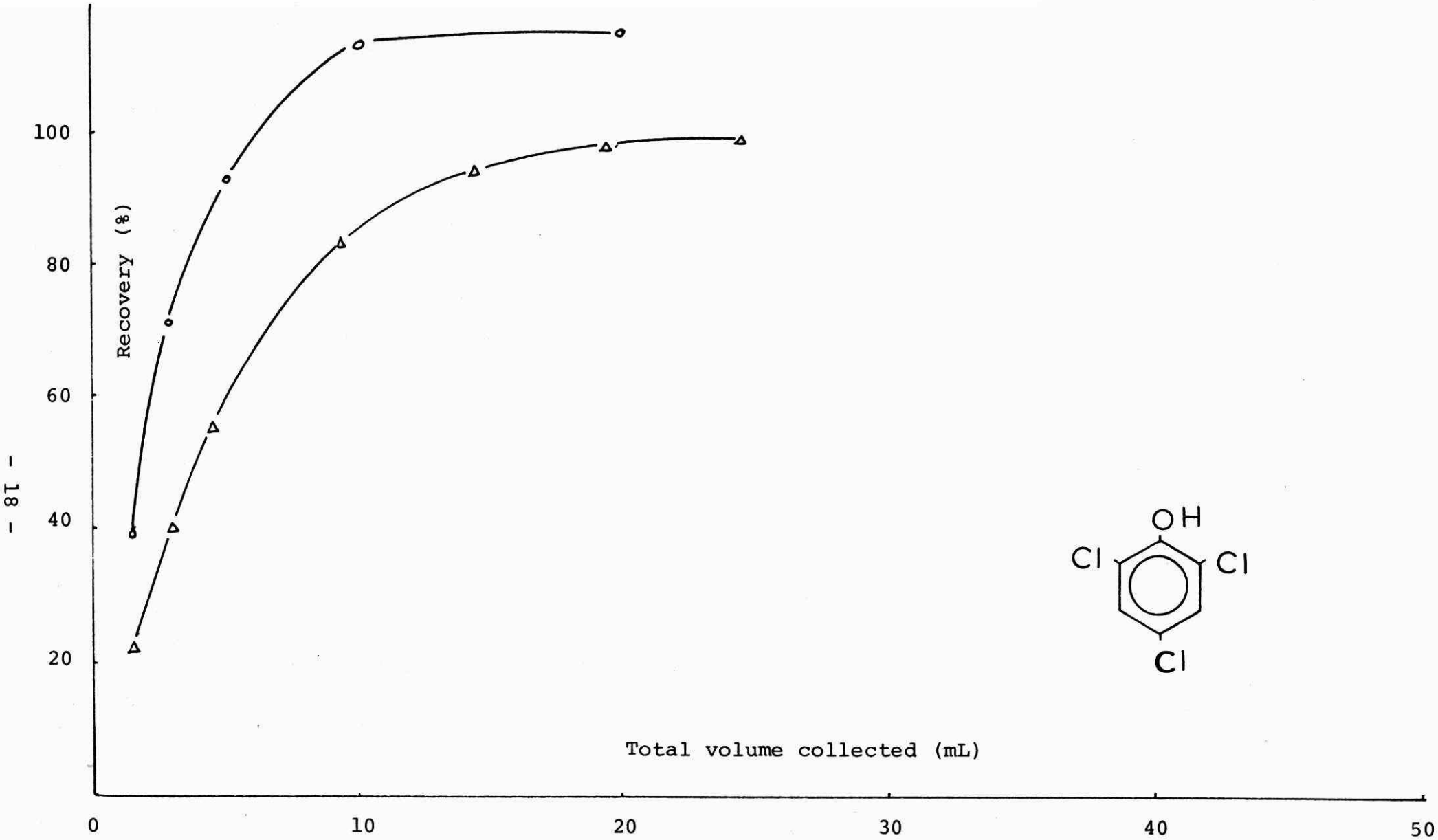


Figure 8. The recovery of 2,4,6-trichlorophenol by steam distillation of a 100 mL solution

∘ Distilled at o.2 mL/min rate, no salt
∆ Distilled at o.5 mL/min rate, no salt

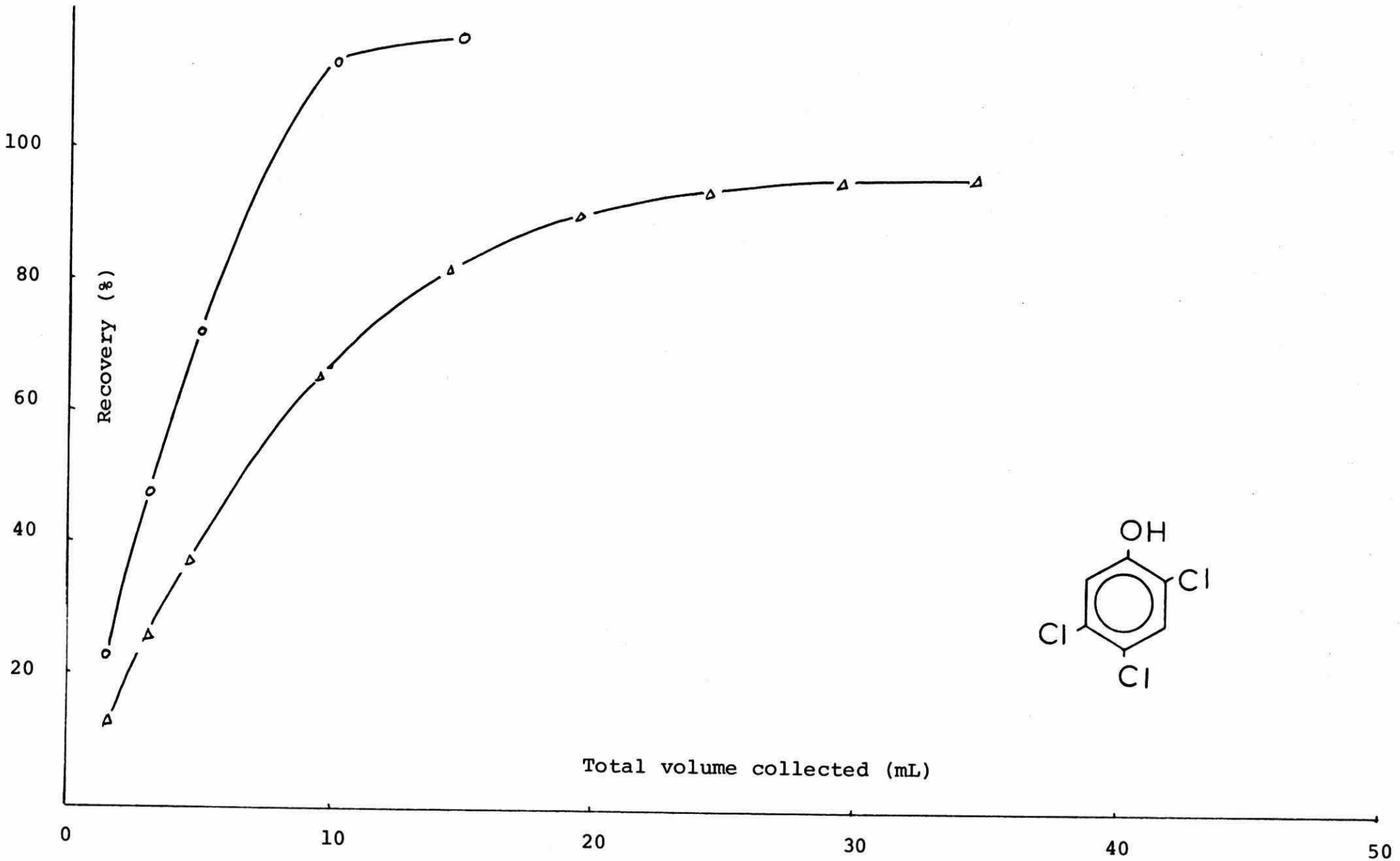


Figure 9. The recovery of 2,4,5-trichlorophenol by steam distillation of a 100 mL solution

o Distilled at o.2 mL/min rate, no salt
Δ Distilled at o.5 mL/min rate, no salt

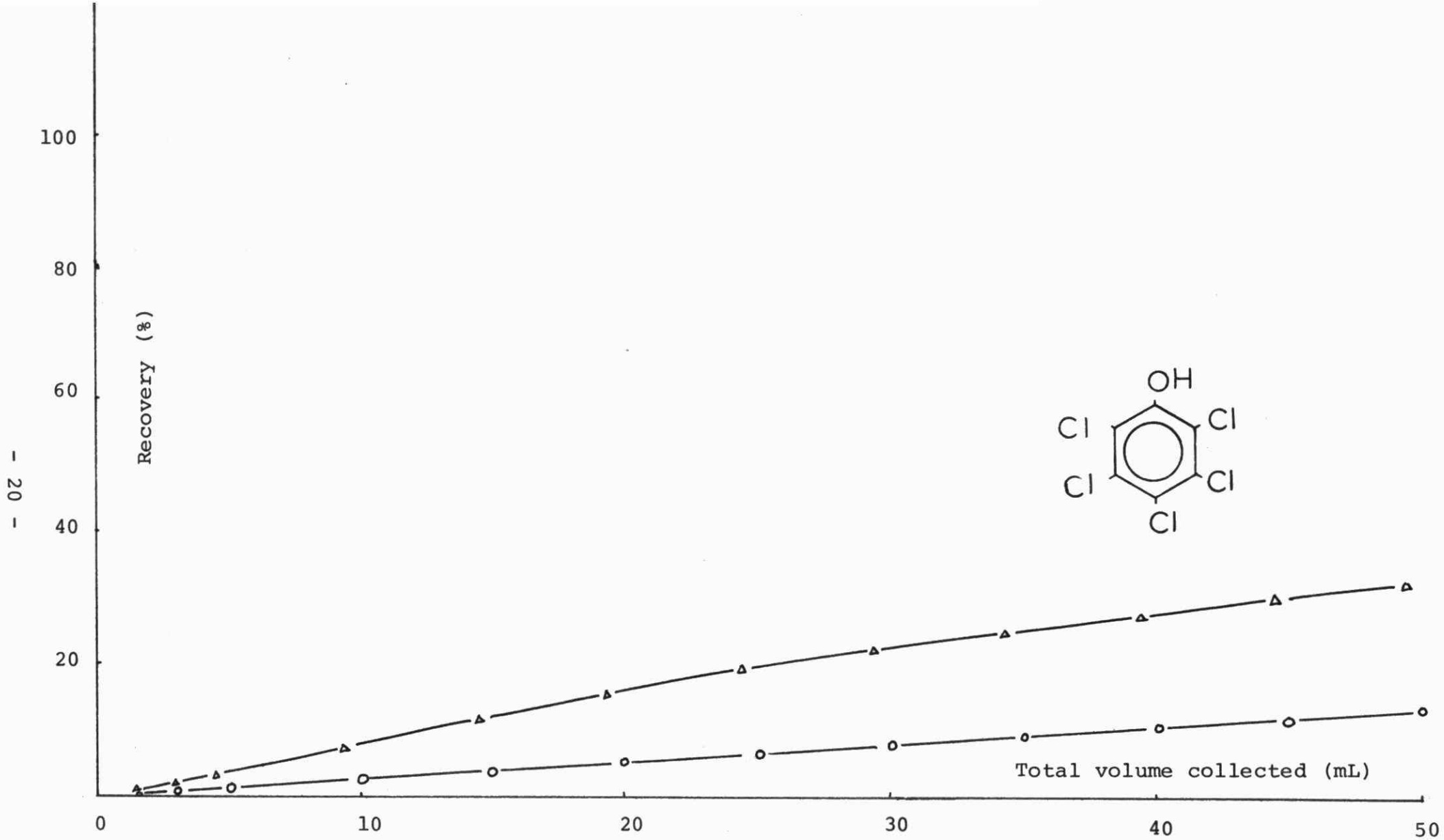


Figure 10. The recovery of pentachlorophenol by steam distillation of a 100 mL solution

o Distilled at 0.2 mL/min rate, no salt
Δ Distilled at o.5 mL/min rate, no salt

Steam Distillation of Phenols
in Dilute Aqueous Solution

Phenol

Distillation: #1
Concentration: 309 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	297	1.4	
2		1.5	754	3.7	(5.1)*
3		2.0	1112	7.2	(12.3)
4		5.0	1038	16.8	(29.1)
5		5.5	822	14.6	(43.7)
6		5.0	544	8.8	(52.5)
7		5.0	522	8.4	(60.9)
8		5.0	451	7.3	(68.2)
9		5.0	396	6.4	(74.6)
10		5.0	340	5.5	(80.1)
11		5.0	297	4.8	(84.9)
12		5.0	229	3.7	(88.6)
13		5.0	185	3.0	(91.6)
14	(Residue)	44.0	62	8.8	

Total Recovered by Steam Distillation	91.6%
Residue	8.8%
GRAND TOTAL	100.4%

* Cumulative values

Steam Distillation of Phenols
in Dilute Aqueous Solution

p-Cresol

Distillation: #1
Concentration: 317 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	613	2.9	
2		1.5	1555	7.4	(10.3)*
3		2.0	2003	12.6	(22.9)
4		5.0	1770	27.9	(50.8)
5		5.5	1088	18.9	(69.7)
6		5.0	556	8.8	(78.5)
7		5.0	451	7.1	(85.6)
8		5.0	359	5.7	(91.3)
9		5.0	293	4.6	(95.9)
10		5.0	209	3.3	(99.2)
11		5.0	144	2.3	(101.5)
12		5.0	90	1.4	(102.9)
13		5.0	48	0.8	(103.7)
14	(Residue)	44.0	ND		

Total Recovered by Steam Distillation	103.7%
Residue	0%
GRAND TOTAL	103.7%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

o-Chlorophenol

Distillation: #1
Concentration: 351 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	7475	31.9	
2		1.5	7360	31.5	(63.4)*
3		2.0	3364	19.2	(82.6)
4		5.0	541	7.7	(90.3)
5		5.5	ND		
6		5.0	ND		
7		5.0	ND		
8		5.0	ND		
9		5.0	ND		
10		5.0	ND		
11		5.0	ND		
12		5.0	ND		
13		5.0	ND		
14	(Residue)	44.0	ND		

Total Recovered by Steam Distillation	90.3%
Residue	0%
GRAND TOTAL	90.3%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

p-Chlorophenol

Distillation: #1
Concentration: 282 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery
1	1.5	114	0.6
2	1.5	282	1.5 (2.1)*
3	2.0	392	2.8 (4.9)
4	5.0	501	8.9 (13.8)
5	5.5	431	8.4 (22.2)
6	5.0	407	7.2 (29.4)
7	5.0	399	7.1 (36.5)
8	5.0	368	6.5 (43)
9	5.0	368	6.5 (49.5)
10	5.0	345	6.1 (55.6)
11	5.0	337	6.0 (61.6)
12	5.0	305	5.4 (67.0)
13	5.0	282	5.0 (72.0)
14 (Residue)	44.0	164	25.6

Total Recovered by Steam Distillation	72.0%
Residue	25.6%
GRAND TOTAL	97.6%

* Cumulative values

Steam Distillation of Phenols
in Dilute Aqueous Solution

2,5-Xylenol

Distillation: #1
Concentration: 281 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1.5	2557	13.6	
2	1.5	3425	18.3	(31.9)*
3	2.0	3391	24.1	(56.0)
4	5.0	1548	27.5	(83.5)
5	5.5	322	6.3	(89.8)
6	5.0	69	1.2	(91.0)
7	5.0	41	0.7	(91.7)
8	5.0	31	0.6	(92.3)
9	5.0	ND		
10	5.0	ND		
11	5.0	ND		
12	5.0	ND		
13	5.0	ND		
14 (Residue)	44.0	ND		

Total Recovered by Steam Distillation	92.3%
Residue	0%
GRAND TOTAL	92.3%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

m-Chloro-p-Cresol

Distillation: #1
Concentration: 336 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	419	1.9	
2		1.5	867	3.9	(5.8)*
3		2.0	11.92	7.0	(12.8)
4		5.0	1300	19.3	(32.1)
5		5.5	976	16.0	(48.1)
6		5.0	661	9.8	(57.9)
7		5.0	602	9.0	(66.9)
8		5.0	537	8.0	(74.9)
9		5.0	488	7.3	(82.2)
10		5.0	412	6.1	(88.3)
11		5.0	347	5.0	(93.3)
12		5.0	249	3.7	(97.0)
13		5.0	217	3.2	(100.2)
14	(Residue)	44.0	54	7.3	

Total Recovered by Steam Distillation	100.2%
Residue	7.3%
GRAND TOTAL	107.5%

* Cumulative values

Steam Distillation of Phenols
in Dilute Aqueous Solution

2,4-Dichlorophenol

Distillation: #1
Concentration: 272 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1.5	6201	34.2	
2	1.5	5603	30.9	(65.1)*
3	2.0	3046	22.4	(87.5)
4	5.0	609	11.2	(98.7)
5	5.5	ND		
6	5.0	ND		
7	5.0	ND		
8	5.0	ND		
9	5.0	ND		
10	5.0	ND		
11	5.0	ND		
12	5.0	ND		
13	5.0	ND		
14 (Residue)	44.0	ND		

Total Recovered by Steam Distillation	98.7%
Residue	0%
GRAND TOTAL	98.7%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

2,4,6-Trichlorophenol

Distillation: #1
Concentration: 317 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1.5	8337	39.4	
2	1.5	6594	31.2	(70.6)*
3	2.0	3487	22.0	(92.6)
4	5.0	1369	21.6	(114.2)
5	5.5	63	1.0	(115.2)
6	5.0	ND		
7	5.0	ND		
8	5.0	ND		
9	5.0	ND		
10	5.0	ND		
11	5.0	ND		
12	5.0	ND		
13	5.0	ND		
14 (Residue)	44.0	ND		

Total Recovered by Steam Distillation	115.2%
Residue	0%
GRAND TOTAL	115.2%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

2,4,5-Trichlorophenol

Distillation: #1
Concentration: 294 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1.5	4512	23	
2	1.5	4867	24.8	(47.8)*
3	2.0	3549	24.1	(71.9)
4	5.0	2494	42.4	(114.3)
5	5.5	162	3.0	(117.3)
6	5.0	ND		
7	5.0	ND		
8	5.0	ND		
9	5.0	ND		
10	5.0	ND		
11	5.0	ND		
12	5.0	ND		
13	5.0	ND		
14 (Residue)	44.0	ND		

Total Recovered by Steam Distillation	117.3%
Residue	0%
GRAND TOTAL	117.3%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

Pentachlorophenol

Distillation: #1
Concentration: 278 ug/L
Volume: 100 mL
Distillation Rate: 0.2 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	69.5	0.4	
2		1.5	69.5	0.4	(0.8)*
3		2.0	69.5	0.4	(1.2)
4		2.0	69.5	1.3	(2.5)
5		5.5	69.5	1.4	(3.9)
6		5.0	69.5	1.3	(5.2)
7		5.0	69.5	1.3	(4.5)
8		5.0	69.5	1.3	(7.8)
9		5.0	69.5	1.3	(9.1)
10		5.0	69.5	1.3	(10.4)
11		5.0	69.5	1.3	(11.7)
12		5.0	69.5	1.3	(13.0)
13		5.0	69.5	1.3	(14.3)
14	(Residue)	44.0	556	88	

Total Recovered by Steam Distillation	14.3%
Residue	88.0%
GRAND TOTAL	102.3%

* Cumulative values

Steam Distillation of Phenols
in Dilute Aqueous Solution

Phenol

Distillation: #2
Concentration: 309 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	464	2.3	
2		1.5	593	2.9	(5.2)*
3		1.5	581	2.8	(8.0)
4		5.0	643	10.4	(18.4)
5		5.0	618	10.0	(28.4)
6		5.0	612	9.9	(38.3)
7		5.0	556	9.0	(47.3)
8		5.0	488	7.9	(55.2)
9		5.0	457	7.4	(62.6)
10		5.0	414	6.7	(69.3)
11		5.0	364	5.9	(75.2)
12		5.0	303	4.9	(80.1)
13		5.0	266	4.3	(84.4)
14	(Residue)	44.0	93	12.0	

Total Recovered by Steam Distillation	84.4%
Residue	12.0%
GRAND TOTAL	96.4%

* Cumulative values

Steam Distillation of Phenols
in Dilute Aqueous Solution

p-Cresol

Distillation: #2
Concentration: 317 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1.5	765	3.6	
2	1.5	945	4.5	(8.1)*
3	1.5	897	4.2	(12.3)
4	5.0	945	14.9	(27.2)
5	5.0	897	14.2	(41.4)
6	5.0	777	12.3	(53.7)
7	5.0	652	10.3	(64.0)
8	5.0	562	8.9	(72.9)
9	5.0	454	7.2	(80.1)
10	5.0	383	6.0	(86.1)
11	5.0	293	4.6	(90.7)
12	5.0	227	3.6	(94.3)
13	5.0	185	2.9	(97.2)
14 (Residue)	44.0	ND		

Total Recovered by Steam Distillation	97.2%
Residue	0%
GRAND TOTAL	97.2%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

o-Chlorophenol

Distillation: #2
Concentration: 351 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	6555	28.0	
2		1.5	4974	21.3	(49.3)*
3		1.5	3709	15.9	(65.2)
4		5.0	1955	27.9	(93.1)
5		5.0	598	8.5	(101.6)
6		5.0	167	2.4	(104)
7		5.0	46	0.7	(104.7)
8		5.0	29	0.4	(105.1)
9		5.0	ND		
10		5.0	ND		
11		5.0	ND		
12		5.0	ND		
13		5.0	ND		
14	(Residue)	40.0	ND		

Total Recovered by Steam Distillation	105.1%
Residue	0%
GRAND TOTAL	105.1%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

p-Chlorophenol

Distillation: #2
Concentration: 282 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	219	1.2	
2		1.5	329	1.8	(3.0)*
3		1.5	313	1.7	(4.7)
4		5.0	376	6.7	(11.4)
5		5.0	392	7.0	(18.4)
6		5.0	392	7.0	(25.4)
7		5.0	368	6.5	(31.9)
8		5.0	384	6.8	(38.7)
9		5.0	360	6.4	(45.1)
10		5.0	352	6.2	(51.3)
11		5.0	329	5.8	(57.1)
12		5.0	305	5.4	(62.5)
13		5.0	298	5.3	(67.8)
14	(Residue)	40.0	219	31.1	

Total Recovered by Steam Distillation	67.8%
Residue	31.1%
GRAND TOTAL	98.9%

* Cumulative values

Steam Distillation of Phenols
in Dilute Aqueous Solution

2,5-Xylenol

Distillation: #2
Concentration: 281 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1.5	1904	10.1	
2	1.5	1945	10.4	(20.5)*
3	1.5	1767	9.4	(29.9)
4	5.0	1466	26.1	(56.0)
5	5.0	973	17.3	(73.3)
6	5.0	589	10.5	(83.8)
7	5.0	349	6.2	(90.0)
8	5.0	199	3.5	(93.5)
9	5.0	110	2.0	(95.5)
10	5.0	75	1.3	(96.8)
11	5.0	48	0.9	(97.7)
12	5.0	34	0.6	(98.3)
13	5.0	27	0.5	(98.8)
14 (Residue)	40.0	27	3.8	

Total Recovered by Steam Distillation	98.8%
Residue	3.8%
GRAND TOTAL	102.6%

* Cumulative values

Steam Distillation of Phenols
in Dilute Aqueous Solution

m-Chloro-p-cresol

Distillation: #2
Concentration: 336 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	520	2.3	
2		1.5	672	3.0	(5.3)*
3		1.5	672	3.0	(8.3)
4		5.0	715	10.6	(18.9)
5		5.0	737	11.0	(29.9)
6		5.0	661	9.8	(39.7)
7		5.0	661	9.8	(49.5)
8		5.0	596	8.9	(58.4)
9		5.0	552	8.2	(66.6)
10		5.0	455	6.8	(73.4)
11		5.0	390	5.8	(79.2)
12		5.0	347	5.2	(84.4)
13		5.0	293	4.4	(88.8)
14	(Residue)	40.0	98	11.7	

Total Recovered by Steam Distillation	88.8%
Residue	11.7%
GRAND TOTAL	100.5%

* Cumulative values

Steam Distillation of Phenols
in Dilute Aqueous Solution

2,4-Dichlorophenol

Distillation: #2
Concentration: 272 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1.5	4026	22.2	
2	1.5	3351	18.5	(40.7)*
3	1.5	2589	14.3	(55.0)
4	5.0	1610	29.6	(84.6)
5	5.0	609	11.2	(95.8)
6	5.0	207	3.8	(99.6)
7	5.0	65	1.2	(100.8)
8	5.0	44	0.8	(101.6)
9	5.0	ND		
10	5.0	ND		
11	5.0	ND		
12	5.0	ND		
13	5.0	ND		
14 (Residue)	40.0	ND		

Total Recovered by Steam Distillation	101.6%
Residue	0%
GRAND TOTAL	101.6%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

2,4,6-Trichlorophenol

Distillation: #2
Concentration: 317 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	4692	22.2	
2		1.5	3855	18.2	(40.4)*
3		1.5	3119	14.8	(55.2)
4		5.0	1826	28.8	(84.0)
5		5.0	685	10.8	(94.8)
6		5.0	216	3.4	(98.2)
7		5.0	63	1.0	(99.2)
8		5.0	ND		
9		5.0	ND		
10		5.0	ND		
11		5.0	ND		
12		5.0	ND		
13		5.0	ND		
14	(Residue)	40.0	ND		

Total Recovered by Steam Distillation	99.2%
Residue	0%
GRAND TOTAL	99.2%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

2,4,5-Trichlorophenol

Distillation: #2
Concentration: 294 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1.5	2616	13.4	
2	1.5	2434	12.4	(25.8)*
3	1.5	2170	11.1	(36.9)
4	5.0	1683	28.6	(65.5)
5	5.0	973	16.6	(82.1)
6	5.0	507	8.6	(90.7)
7	5.0	203	3.5	(94.2)
8	5.0	91	1.6	(95.8)
9	5.0	41	0.7	(96.5)
10	5.0	ND		
11	5.0	ND		
12	5.0	ND		
13	5.0	ND		
14 (Residue)	40.0	ND		

Total Recovered by Steam Distillation	96.5%
Residue	0%
GRAND TOTAL	96.5%

* Cumulative values
ND = Not Detected

Steam Distillation of Phenols
in Dilute Aqueous Solution

Pentachlorophenol

Distillation: #2
Concentration: 278 ug/L
Volume: 100 mL
Distillation Rate: 0.5 mL/min

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1.5	209	1.1	
2		1.5	226	1.2	(2.3)*
3		1.5	243	1.3	(3.6)
4		5.0	226	3.9	(7.5)
5		5.0	243	4.2	(11.7)
6		5.0	226	3.9	(15.6)
7		5.0	226	3.9	(19.5)
8		5.0	174	3.0	(22.5)
9		5.0	156	2.7	(25.2)
10		5.0	156	2.7	(27.9)
11		5.0	174	3.0	(30.9)
12		5.0	122	2.1	(33.0)
13		5.0	122	2.1	(35.1)
14	(Residue)	40.0	591	82.4	

Total Recovered by Steam Distillation	35.1%
Residue	82.4%
GRAND TOTAL	117.5%

* Cumulative values

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

Phenol

Distillation: #3
Distillation Rate: 0.2 mL/min
Concentration: 300 ug/L
Volume: 100 mL

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1	1250	4.2	
2	1	3940	13.1	(17.3)*
3	1	4500	15.0	(32.3)
4	5	2150	35.8	(68.1)
5	5	255	4.3	(72.9)
6	5	ND		
7	5	ND		
8	5	ND		
9	5	ND		
10	5	ND		
11	5	ND		
12	5	ND		
13	5	ND		
14 (Residue)	NA	NA		

Total Recovered by Steam Distillation 72.4%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

p-Cresol

Distillation: #3

Distillation Rate: 0.2 mL/min
Concentration: 284 ug/L
Volume: 100 mL

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1	4800	16.9	
2	1	5200	18.3	(35.2)*
3	1	4300	15.1	(50.3)
4	5	938	16.5	(66.8)
5	5	163	2.9	(69.7)
6	5	ND		
7	5	ND		
8	5	ND		
9	5	ND		
10	5	ND		
11	5	ND		
12	5	ND		
13	5	ND		
14 (Residue)	NA	NA		

Total Recovered by Steam Distillation 69.2%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

p-Chlorophenol

Distillation: #3

Distillation Rate: 0.2 mL/min
Concentration: 338 ug/L
Volume: 100 mL

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1	1650	4.9	
2	1	2500	7.4	(12.3)*
3	1	2600	7.7	(20.0)
4	5	2150	31.8	(51.8)
5	5	1375	20.3	(72.1)
6	5	305	4.5	(76.6)
7	5	ND		
8	5	ND		
9	5	ND		
10	5	ND		
11	5	ND		
12	5	ND		
13	5	ND		
14 (Residue)	NA	NA		

Total Recovered by Steam Distillation 76.6%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

2,5-Xylenol

Distillation: #3

Distillation Rate: 0.2 mL/min
Concentration: 250 ug/L
Volume: 100 mL

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1	12000	48	
2	1	4600	18.4	(66.4)*
3	1	1300	5.2	(71.6)
4	5	150	3.0	(74.6)
5	5	110	2.2	(76.8)
6	5	ND		
7	5	ND		
8	5	ND		
9	5	ND		
10	5	ND		
11	5	ND		
12	5	ND		
13	5	ND		
14 (Residue)	NA	NA		

Total Recovered by Steam Distillation 76.8%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

p-Chloro-m-cresol

Distillation: #3

Distillation Rate: 0.2 mL/min
Concentration: 448 ug/L
Volume: 100 mL

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1	8200	18.3	
2	1	5250	11.7	(30.0)*
3	1	4550	10.2	(40.2)
4	5	2525	28.2	(68.4)
5	5	400	4.5	(72.9)
6	5	ND		
7	5	ND		
8	5	ND		
9	5	ND		
10	5	ND		
11	5	ND		
12	5	ND		
13	5	ND		
14 (Residue)	NA	NA		

Total Recovered by Steam Distillation 72.9%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

Phenol

Distillation: #4

Distillation Rate: 0.5 mL/min
Concentration: 300 ug/L
Volume: 100 mL

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1	2500	8.3	
2		1	3075	10.3	(18.6)*
3		1	3125	10.4	(29.0)
4		5	2000	33.3	(62.3)
5		5	260	4.3	(66.6)
6		5	ND		
7		5	ND		
8		5	ND		
9		5	ND		
10		5	ND		
11		5	ND		
12		5	ND		
13		5	ND		
14	(Residue)	NA	NA		

Total Recovered by Steam Distillation 66.7%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

p-Cresol

Distillation: #4

Distillation Rate: 0.5 mL/min
Concentration: 284 ug/L
Volume: 100 mL

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1	3000	10.6	
2		1	3550	12.5	(231)*
3		1	6200	21.8	(49.9)
4		5	1350	23.8	(68.7)
5		5	50	0.9	(69.6)
6		5	ND		
7		5	ND		
8		5	ND		
9		5	ND		
10		5	ND		
11		5	ND		
12		5	ND		
13		5	ND		
14	(Residue)	NA	NA		

Total Recovered by Steam Distillation 69.6%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

p-Chlorophenol

Distillation: #4

Distillation Rate: 0.5 mL/min
Concentration: 338 ug/L
Volume: 100 mL

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1	650	1.9	
2	1	1250	3.7	(5.6)*
3	1	2200	6.5	(12.1)
4	5	1950	28.8	(40.9)
5	5	1000	14.8	(55.7)
6	5	340	5.0	(60.7)
7	5	185	2.7	(63.4)
8	5	50	0.7	(64.1)
9	5	ND		
10	5	ND		
11	5	ND		
12	5	ND		
13	5	ND		
14 (Residue)	NA	NA		

Total Recovered by Steam Distillation 64.3%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

2,5-Xylenol

Distillation: #4

Distillation Rate: 0.5 mL/min
Concentration: 250 ug/L
Volume: 100 mL

Fraction #		Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1		1	12000	48	
2		1	4600	18.4	(66.4)*
3		1	575	2.3	(68.7)
4		5	150	3.0	(71.7)
5		5	110	2.2	(73.9)
6		5	ND		
7		5	ND		
8		5	ND		
9		5	ND		
10		5	ND		
11		5	ND		
12		5	ND		
13		5	ND		
14	(Residue)	NA	NA		

Total Recovered by Steam Distillation 73.9%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols
in Dilute Salt Saturated Aqueous Solution

P-Chloro-m-cresol

Distillation: #4

Distillation Rate: 0.5 mL/min
Concentration: 448 ug/L
Volume: 100 mL

Fraction #	Fraction Vol. (mL)	Fraction Conc. ug/L	% Recovery	
1	1	4600	10.3	
2	1	6650	14.8	(25.1)*
3	1	7000	15.6	(40.7)
4	5	2500	27.9	(68.6)
5	5	238	2.7	(71.3)
6	5	ND		
7	5	ND		
8	5	ND		
9	5	ND		
10	5	ND		
11	5	ND		
12	5	ND		
13	5	ND		
14 (Residue)	NA	NA		

Total Recovered by Steam Distillation 71.3%

* Cumulative values
ND = Not Detected
NA = Not Analyzed

Steam Distillation of Phenols

Recoveries in the First 30 mL (30%) of
Distillates at two Distillation Rates

Phenols	Recoveries (%)			
	No Salt		Salt Saturated*	
	0.2 mL/min	0.5 mL/min	0.2 mL/min	0.5 mL/min
Phenol	65.1	55.2	72.4	66.7
p-Cresol	86.5	72.9	69.2	69.6
o-Chlorophenol	90.3	105.1	NT	NT
p-Chlorophenol	41.5	38.7	76.6	60.9
2,5-Xylenol	92.3	93.5	76.8	73.9
m-Chloro-p-cresol	71.7	58.4	72.9	71.3
2,4-Dichlorophenol	98.7	95.8	NT	NT
2,4,6-Trichlorophenol	115.2	98.2	NT	NT
2,4,5-Trichlorophenol	117.3	96.5	NT	NT
Pentachlorophenol	9.2	22.5	NT	NT

* See page 9 for explanation

NT = Not Tested

Steam Distillation of Phenols

Recoveries in the First 50 mL (30%) of
Distillates at two Distillation Rates

Phenols	Recoveries (%)			
	No Salt		Salt Saturated*	
	0.2 mL/min	0.5 mL/min	0.2 mL/min	0.5 mL/min
Phenol	88	80	72.4	66.7
p-Cresol	102.9	94.3	69.2	69.6
o-Chlorophenol	90.3	105.1	NT	NT
p-Chlorophenol	67.0	62.5	76.6	60.9
2,5-Xylenol	92.3	98.3	76.8	73.9
m-Chloro-p-cresol	97.0	84.4	72.9	71.3
2,4-Dichlorophenol	98.7	101.6	NT	NT
2,4,6-Dichlorophenol	115.2	99.2	NT	NT
2,4,5-Dichlorophenol	117.3	96.5	NT	NT
Pentachlorophenol	13	33.0	NT	NT

* See page 9 for explanation

NT = Not Tested

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